Bench Scale Development and Testing of Aerogel Sorbents for CO_2 Capture

Award No. DE-FE0013127

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Subcontractors: ADA-ES, Inc., University of Akron

Start Date: October 1, 2013

BP1 End Date: September 30, 2014

BP2 End Date: September 30, 2015

BP3 End Date: September 30, 2016

NANOTECHNOLOGY AT WORK

Agenda

I. Introduction
- Aspen Aerogels, Inc.
- Aerogels
- Background into the Technologies for CO_2 Capture
- ADA Environmental Solutions
- Results Summary of SBIR Phase II Program (DE-SC0004289)

II. Cooperative Agreement Program Overview
- Objective
- Team
- Tasks
- Schedule
- Milestones
- Deliverables
- Current Status

Aspen Aerogels, Inc.

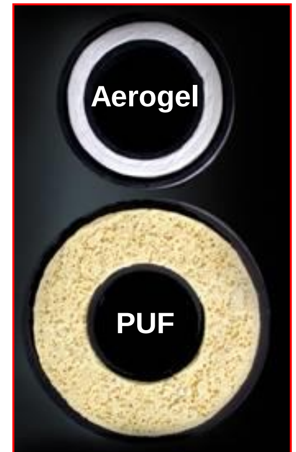
- Founded in 2001
- Privately owned
- 210 Employees
- Locations
  - Northborough, MA
    - *(headquarters, R&D laboratories)*
  - East Providence, RI
    - *(manufacturing facility)*
- Current Capacity > 50 million sq.ft./yr.
- World's leading manufacture of flexible aerogel blankets
- ISO 9001-2000 (BVQi certified)

Aerogel Timeline

Aerogels invented

Aerogels for spacesuits

Aspen makes fiber-reinforced aerogels



Aerogels offshore

Aerogels for the petrochemical processing industry

1930s | 1993 | 1999 | 2001 | 2003 | 2005 | 2007 | 2013

From the 30's to the 80's, many large chemical companies tried to produce aerogels

Aspen Aerogels born

Plant 1 opens

7-10 MM sqft capacity

Plant 2 opens

30-100 MM sqft capacity

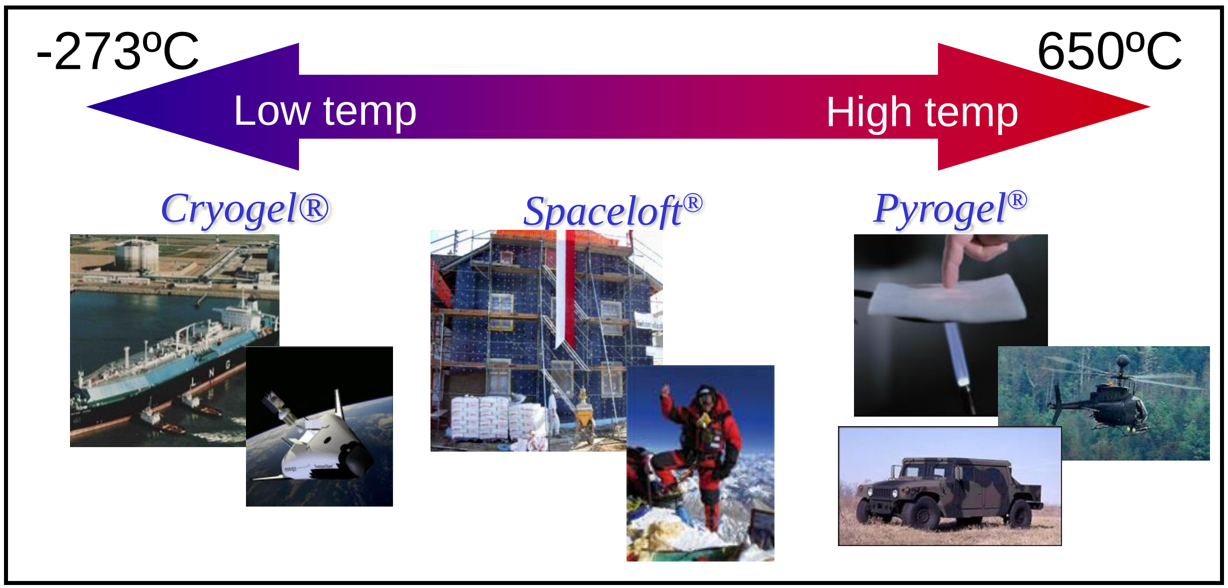
Aerogels in the Mainstream – Thermal Insulation

Two Aspen innovations moved aerogels from lab curiosity to high-volume industrial product:

1. Aspen's supercritical CO_2 extraction process reduces cycle time from months to hours
2. Casting the wet gel into a fibrous batting provides mechanical integrity

Aerogel Monolith

Aerogel Blankets

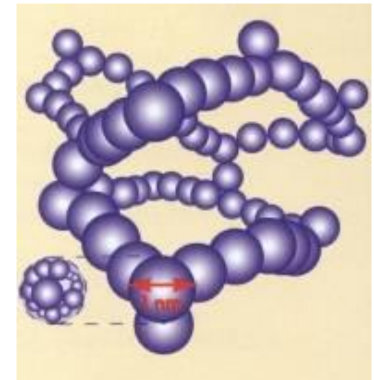


<u>The Aspen Advantage</u>:
Superinsulation performance in a flexible blanket form

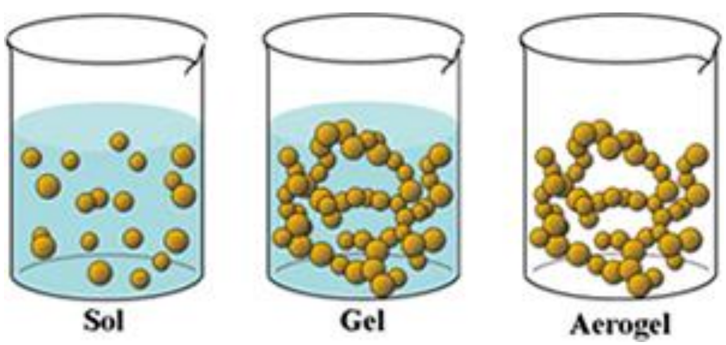
What are Aerogels?

Nanoporous solid with a specific structural morphology……

- Open structure - up to 99% open porosity
- Pore diameters = ~ 10 nm average
  (< 1/30,000th the width of a human hair)
- Nanoporosity slows heat and mass transport, providing record-low thermal conductivity



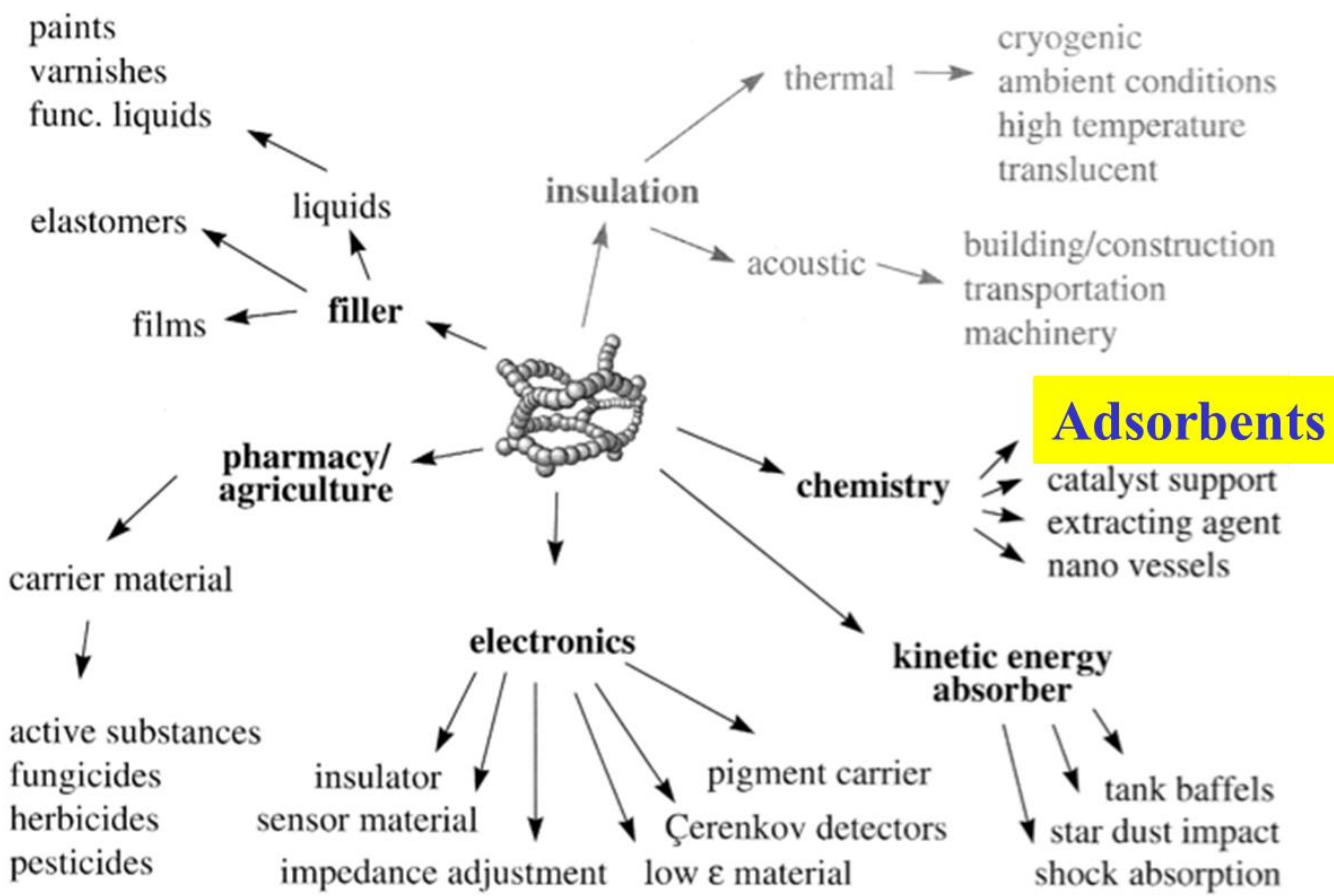
$Si(OR)_4 + 2\ H_2O \rightarrow (SiO_2)_x + 4\ ROH$



…..and method of production

1. Sol-gel Process
2. Aging Process
3. Extraction Process
4. Drying Process

Aerogel Applications



http://uspace.shef.ac.uk/docs/DOC-51449

Background into the Technologies for CO_2 Capture

CO_2 Capture technologies:

- ***Liquid sorbent*** (MEA), more mature

- ***Membranes*** with high permeances and high CO_2/N_2 selectivities have been developed.

> **Challenge:** the scale of the process and the very large, expensive, and energy-consuming compression equipment needed.

- ***Solid sorbents*** more efficient, mainly amine-functionalized silica, Carbon, zeolites, MOFs, …

> **Challenges:** sorbent cyclic stability, capacity (economics)

Background into the Technologies for CO_2 Capture

<u>Aqueous amines circulate 70% H_2O</u>

- ▼ Heating the inert water is energy intensive technology ~3600 kJ/kgCO_2
- ▼ High corrosion & maintenance costs

<u>Solid sorbents</u>

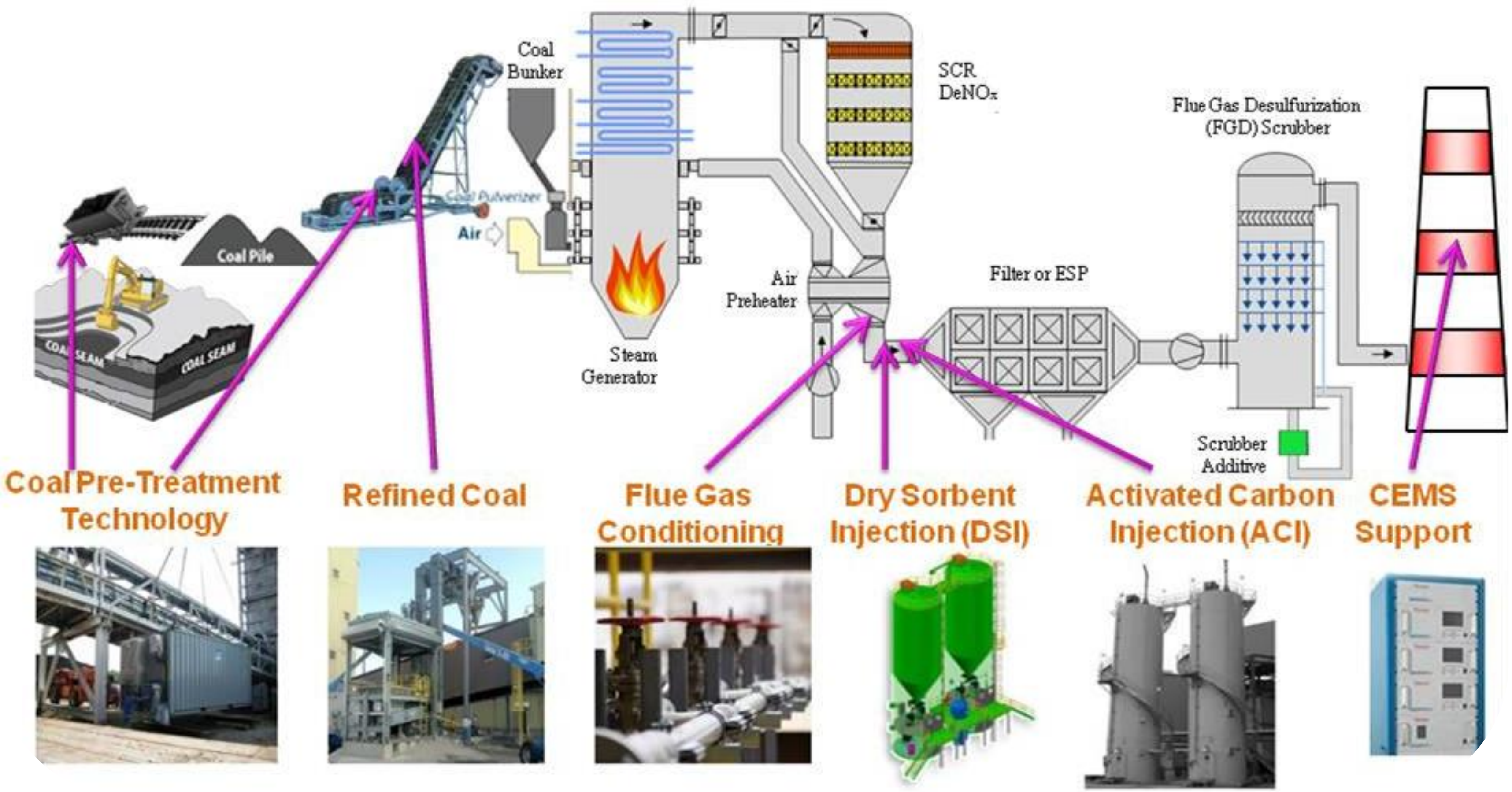
- ▲ Promise to lower regeneration energy (not circulating water)
- ▼ Sorbent attrition

<u>Hydrophobic Amine Functionalized Aerogel (AFA)</u>

- ▲ Amine groups bonded to aerogel backbone (cyclic stability)
- ▲ Hydrophobic aerogel - Stable in liquid & high moisture atmospheres
- ▲ Promise to lower regeneration energy (not circulating water)
- ▼ Sorbent attrition

ADA Environmental Solutions:

Providing clean technology for the coal-fired industry



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Commercial Technology Needs

- Reduce parasitic energy requirements
- Minimize water usage and disposal issues
- Minimize life-cycle impacts
  - Raw materials
  - Fate of waste
- Minimize space requirements for installed process
- Allow flexible plant operations
  (fossil plants follow availability of
  renewable power)
- Minimize capital and operating costs
- Adaptable for progressive CO_2 emissions requirements

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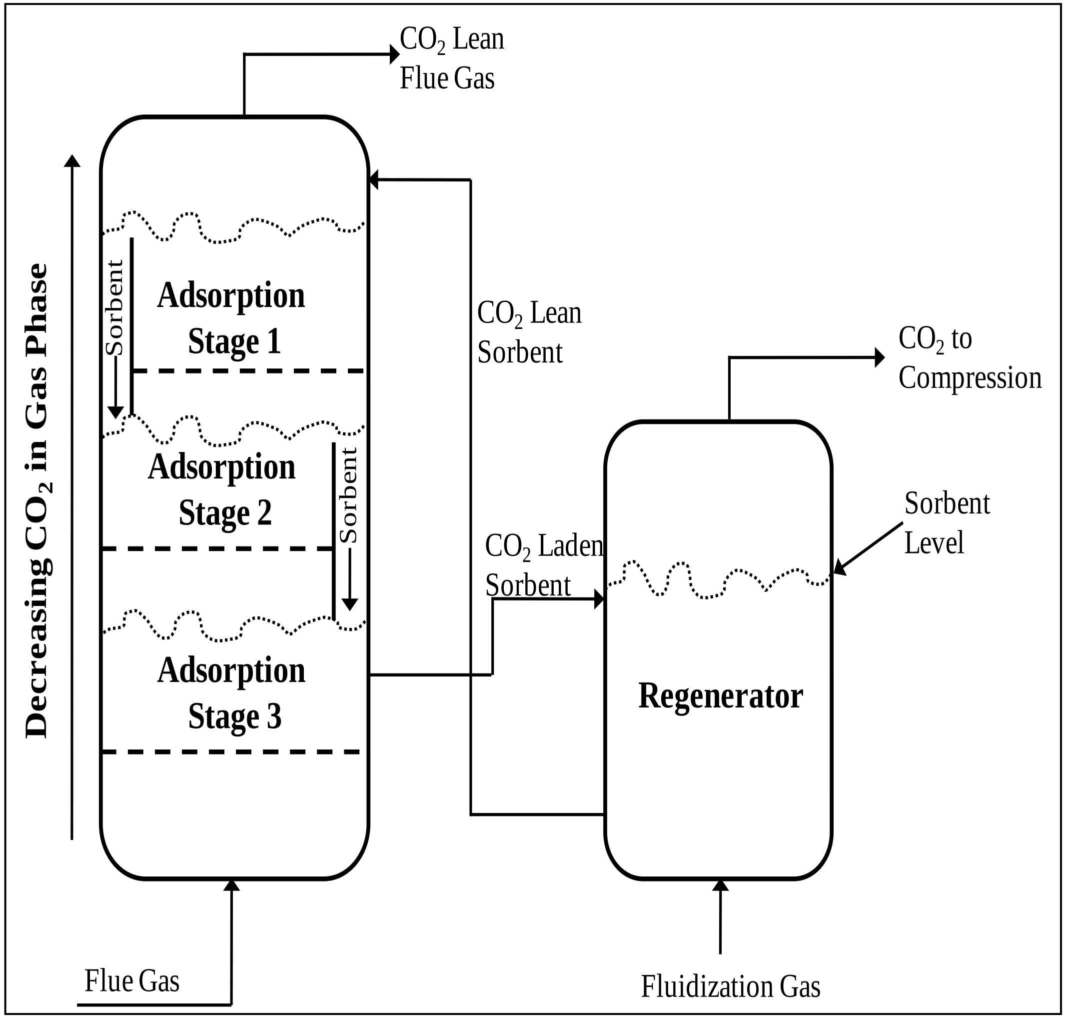
ADAsorb™ Process Overview

- Flue gas passes through adsorber module where sorbent particle adsorbs CO_2
- Regenerable solid sorbent cycles between adsorber and regenerator.
- Increased temperature in regenerator releases CO_2



Results Summary of Phase II SBIR Program

Award No. DE-SC0004289

Phase II SBIR Objectives

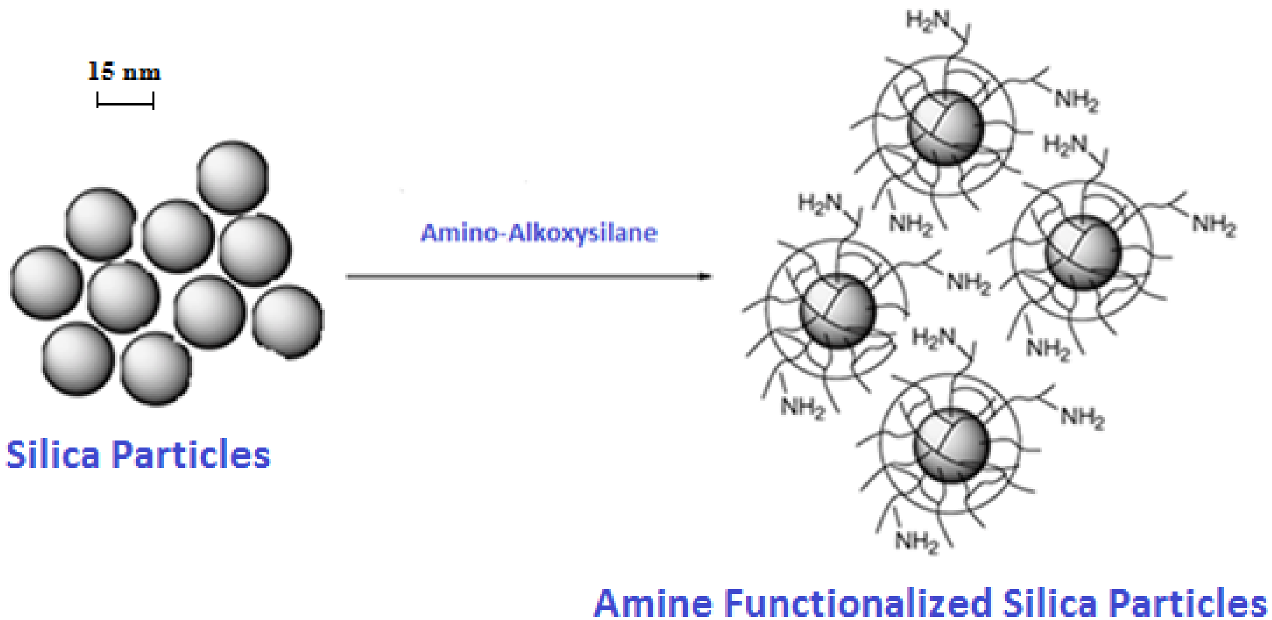
1) Maximize CO_2 working capacity of AFA.
2) Develop sorbent synthesis concepts for production.
3) Demonstrate longer cycle life of AFA sorbents (>2000 cycles).
4) Characterize the impact of SO_x and NO_x contaminants.
5) Pelletize the best performing AFA sorbent.
6) Evaluate hydrodynamic, thermal properties on fluidized bed (4 ft^3).

Phase II SBIR Accomplishments

Test Parameter	Target	Achieved
CO_2 adsorption capacity	> 12 wt.%	22 wt.%
CO_2 working capacity	> 5 wt.%	6.4 - 9 wt.%
Adsorb/Desorb stability	Stable >2,000 cycles	Stable >2,000 cycles
Thermal stability	>120°C	>130°C
Low regeneration energy	< 2400 kJ/kgCO_2	~ 2000 kJ/kgCO_2 *
Resistance to SO_X and NO_X	50ppmv, 80ppmv	100ppmv, 100ppmv

* Estimated

Phase II SBIR Aerogel Optimization



- More than 200 AFAs were synthesized by sol-gel
- Amine distributed throughout silica backbone structure, not surface coating
- Different types of amine precursors (mono-amine, polyamine, polyimine) used.
- Two methods of impregnation/grafting were investigated.
- Silica matrix is based on methyl-silicate (hydrophobic) aerogel.

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Phase II SBIR Aerogel Sorbent Testing

Key Sorbent Characterization Tests

- Technical Assessment
  - CO_2 loading over range of T&P
  - H_2O loading at select T&P
  - Sorbent kinetics for CO_2 and moisture
  - Sorbent selectivity for CO_2
  - Longer-term CO_2 capture stability
  - Relative Attrition
  - Optimal particle size distribution for fluidized bed
  - Hydrodynamic properties

- → **Economic Assessment**

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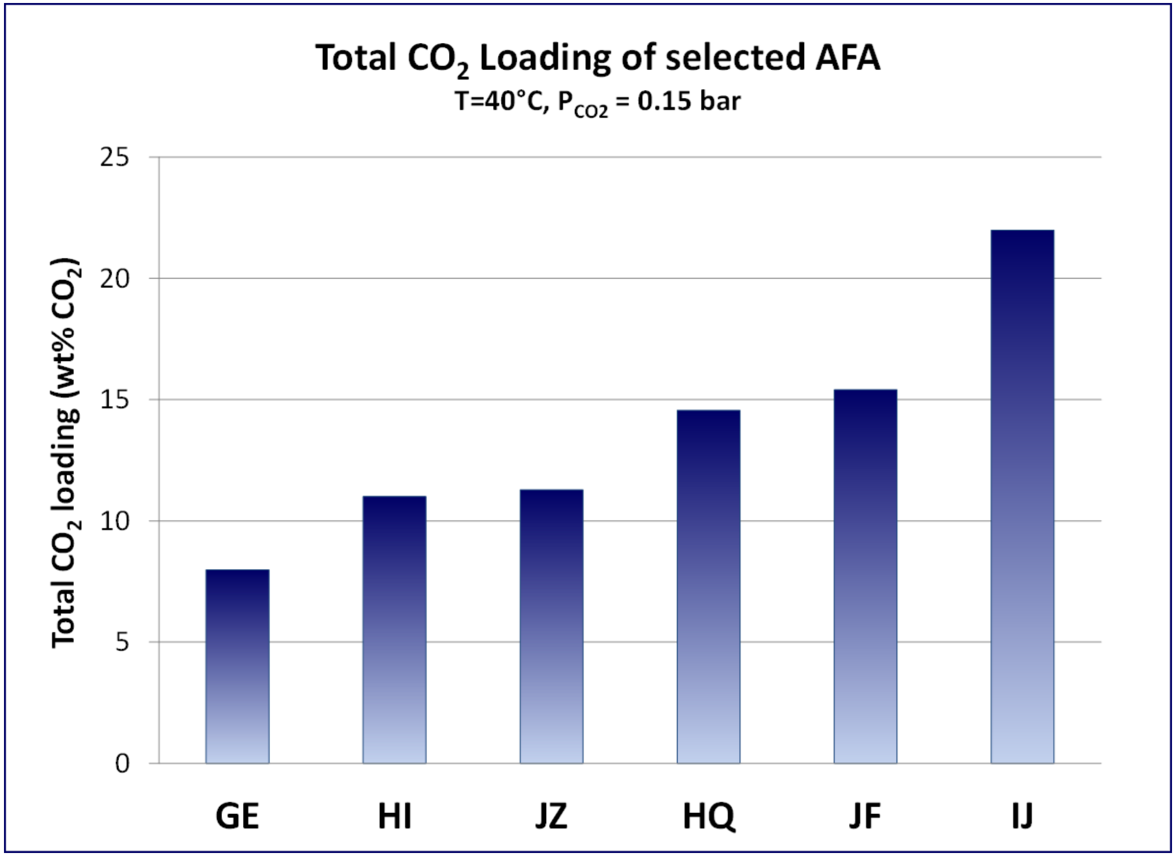
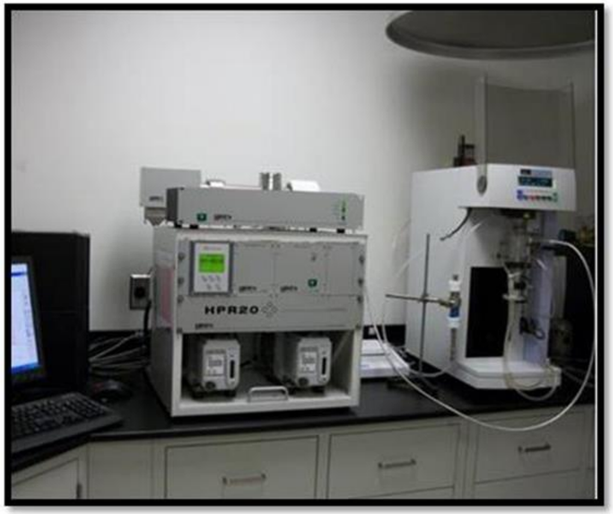
Phase II SBIR Results

Total CO_2 Loading





TGA and Mass Spec

AFA sorbent performance is a function of the sol-gel process conditions: nature of amine precursor, amine loading, sorbent density

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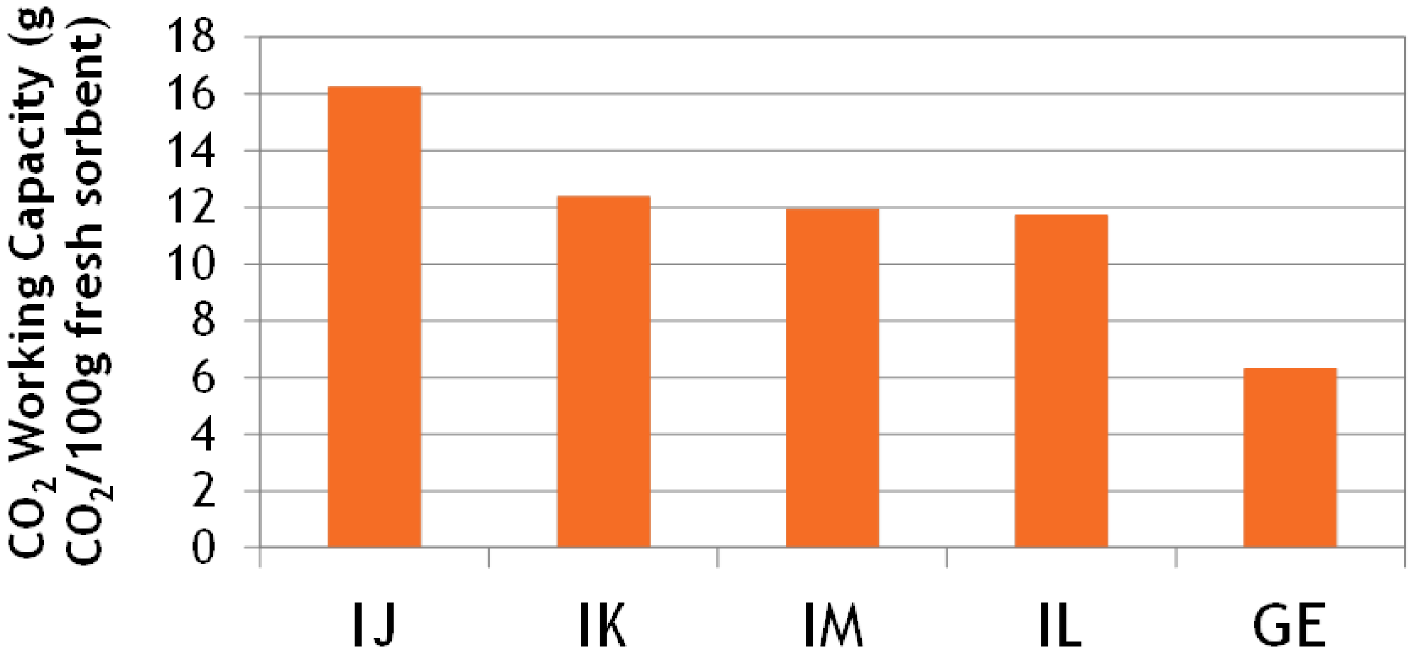
Phase II SBIR Results

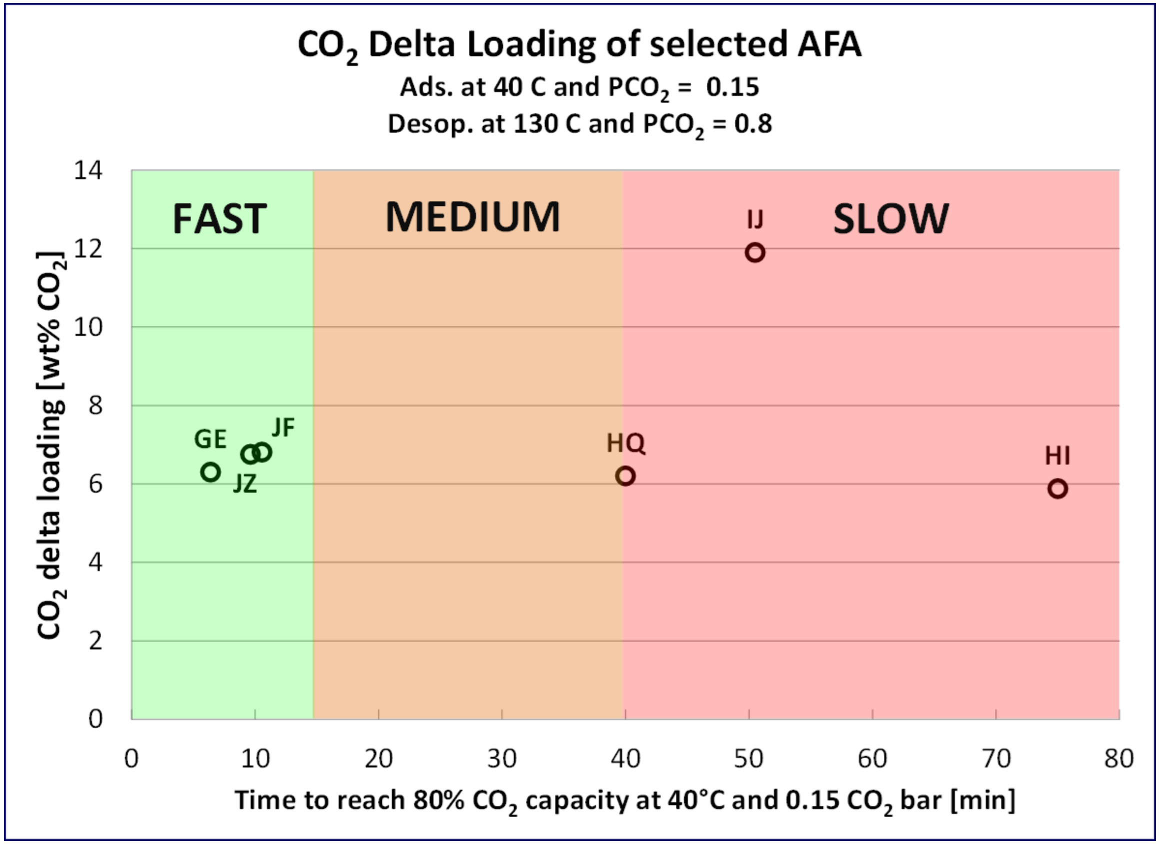
CO_2 "working" capacity



- Greater "working capacity" reduces required sorbent quantity, equipment size, and process costs

Phase II SBIR Results

Adsorption/desorption Kinetics



AFA sorbent performance is a function of the sol-gel process conditions: nature of amine precursor, amine loading, sorbent density

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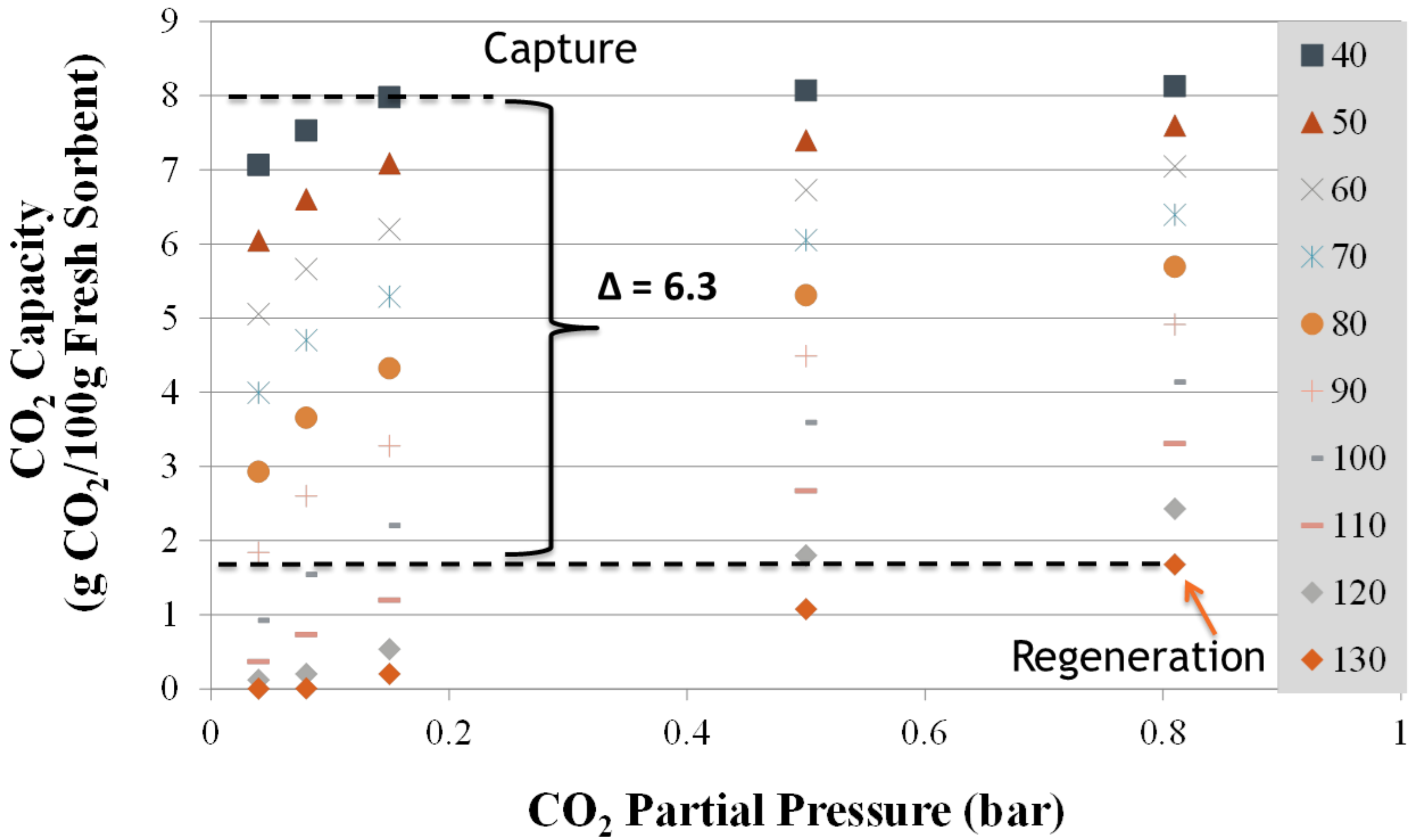
Phase II SBIR Results

CO_2 Isotherms : GE Powder



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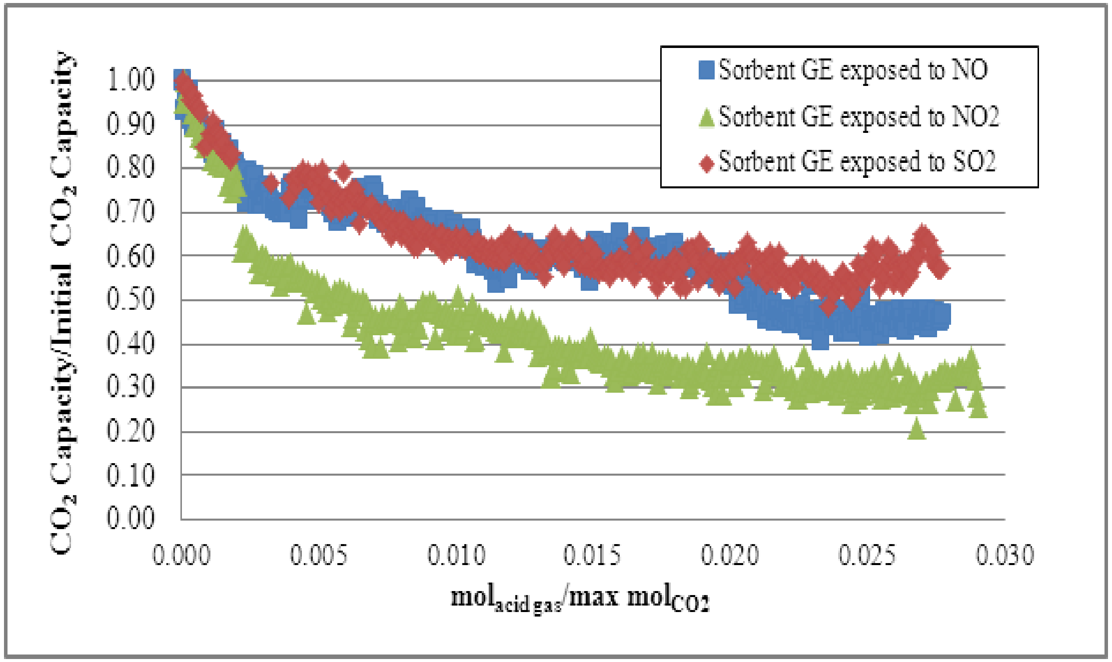
Phase II SBIR Results

Potential AFA resistance to SO_x and NO_x

- The CO_2 capacity of sorbent GE was evaluated with simulated flue gas that included acid gases (either NO, NO_2, or SO_2) at a concentration of 100 ppm.

- 400 cycles were completed for each test.

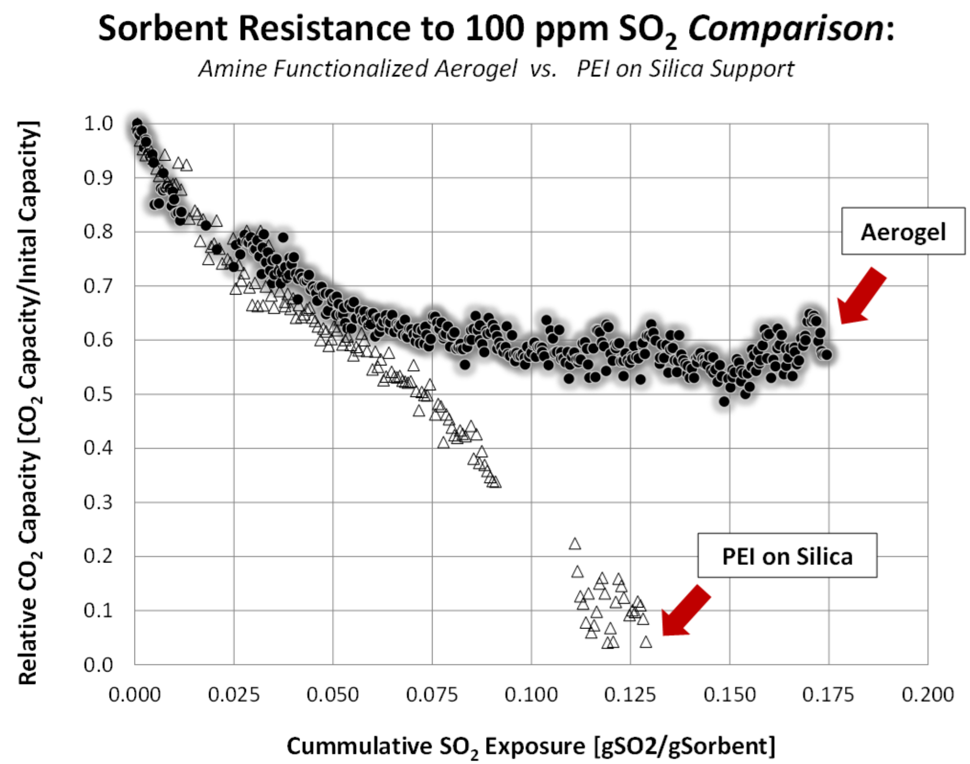


GE is more tolerant of acid gases than what has been observed for other sorbents tested by ADA

Phase II SBIR Results

Comparison of Sorbent resistance to SO_x

AFAs demonstrated much better resistance to CO_2 capacity degradation from 100 ppm SO_2 than control, PolyEthyleneImine (PEI) on silica support



After 400 adsorption/desorption cycles, AFA sorbent retains 60% of its initial CO_2 capacity, whereas the PEI/silica retains < 5% of its initial capacity.

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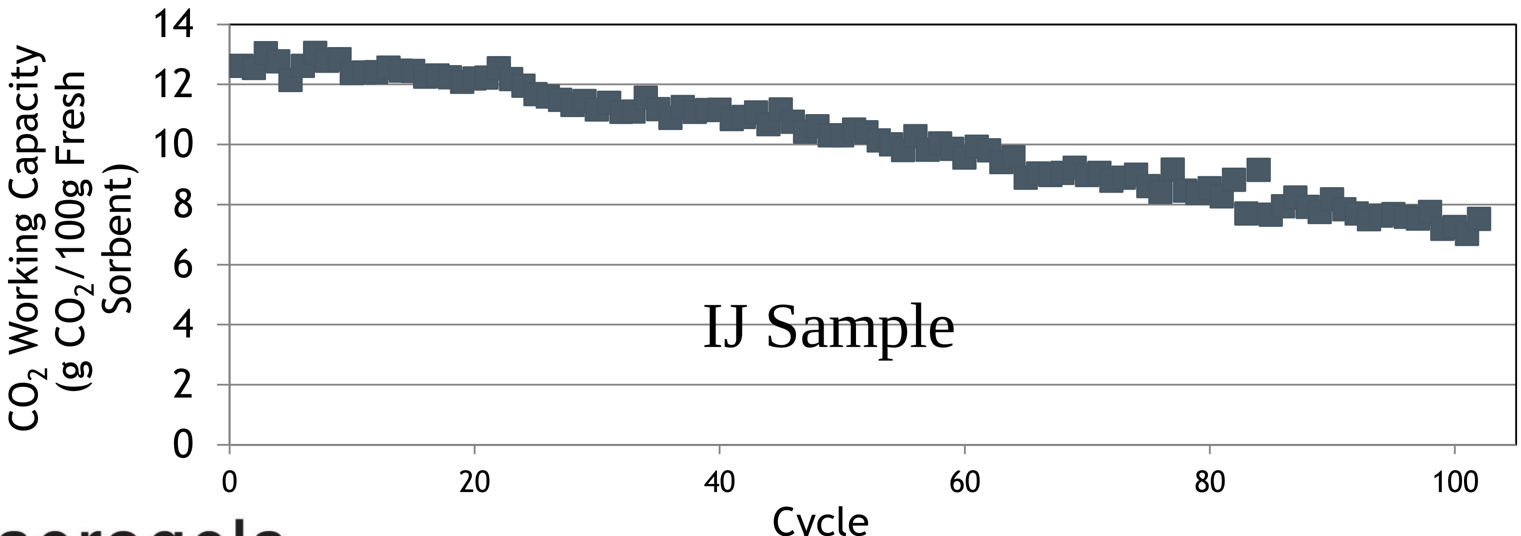
AFA Long Term Stability

- Top HQ-type sorbents evaluated for cyclic stability
  - Adsorption at P_{CO2}=0.15, 40°C
  - Regeneration at P_{CO2} = 0.81, 130°C
- Performance degraded over time

Automated Fixed Bed



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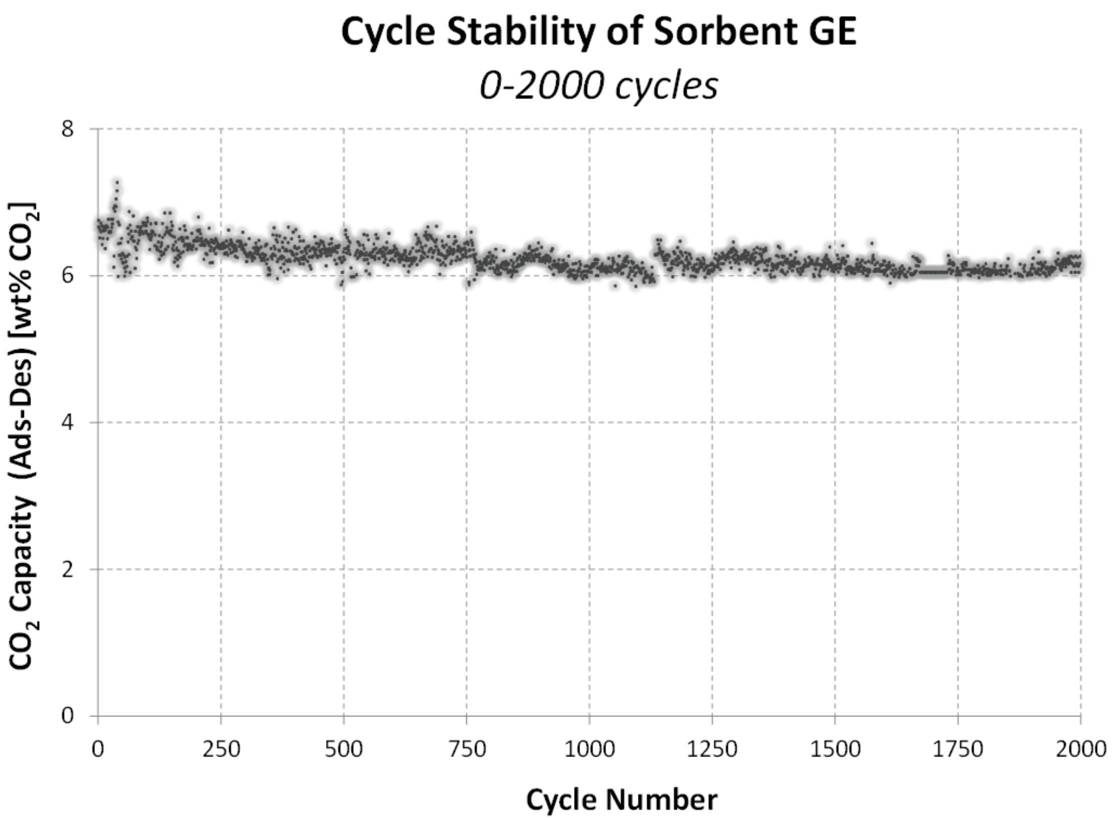
Phase II SBIR Results



Sorbent GE – lower initial capacity

6% loss of initial CO_2 delta capacity after 2,000 cycles

Stability is superior to any supported amine CO_2 sorbent evaluated by ADA-ES to date

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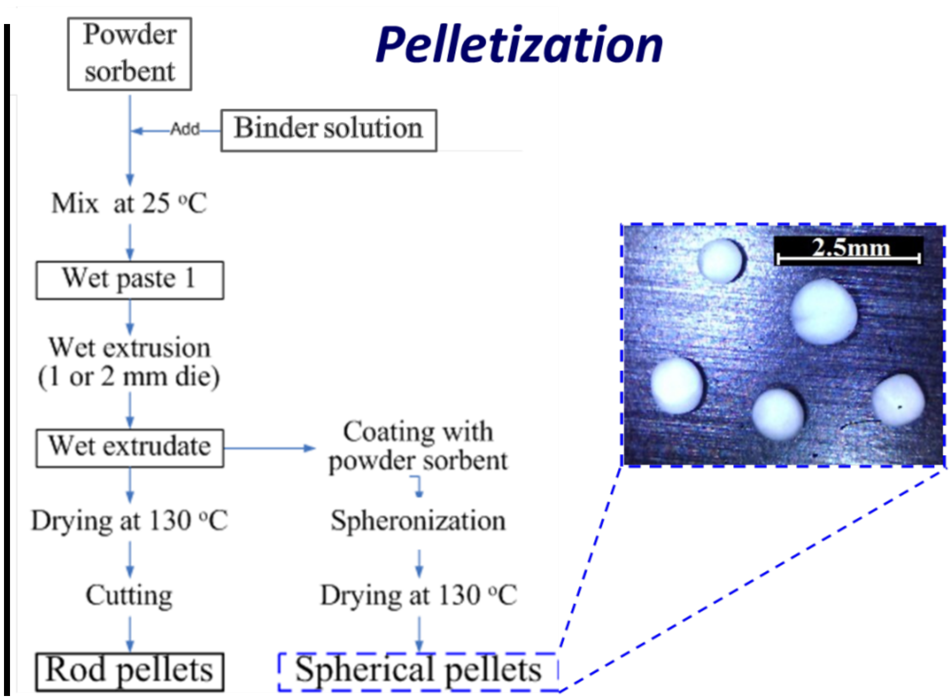
Phase II SBIR Results

AFA Sorbent Pellets

University of Akron developed polymeric binders that cause agglomeration of sorbent particles and provide linkages that allow spacing for CO_2 diffusion.

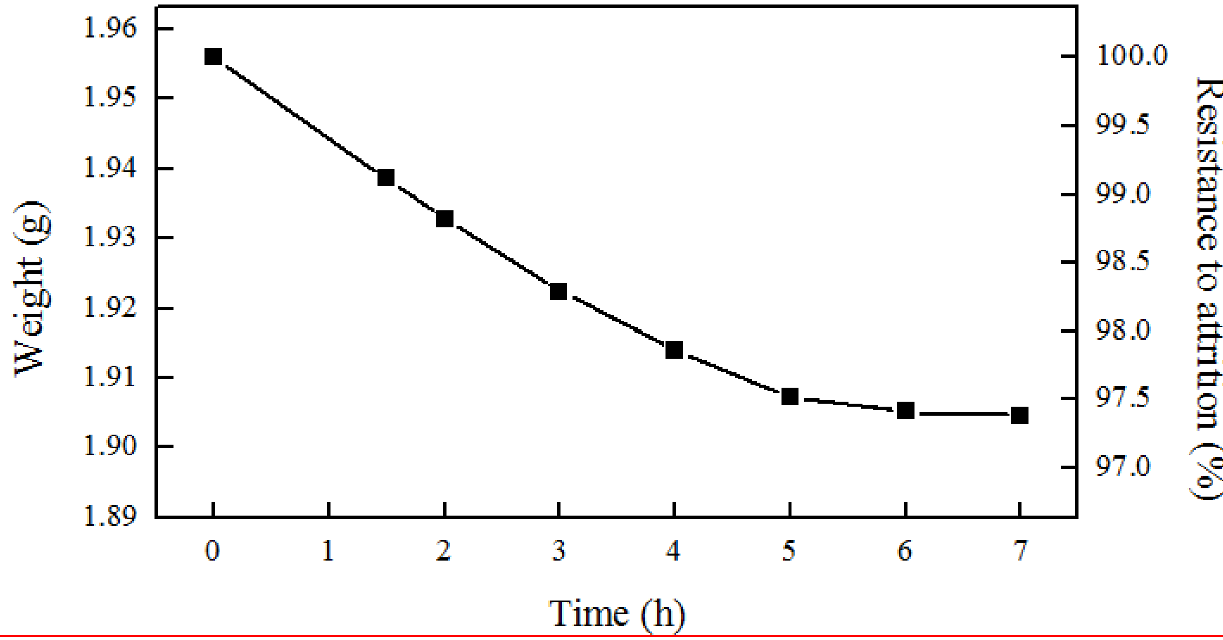
Sorbents	Binder/Sorbent ratio	Pellet Strenght	CO_2 capture capacity (mmol CO_2/g-sorbent)
GE	0	_	2.11
GE	1:1	Strong	2.23
GE	2:1	Strong	2.45
GE	2:3	Strong	1.88
HQ	0	_	2.30
HQ*	1:1	Weak	
JZ	0	_	2.53
JZ	1:1	Strong	0.25
UA standard	0	_	2.80

*difficult to pelletize
CO_2 capture capacity measured at Akron. Units in mmol, not wt%)



Phase II SBIR Results

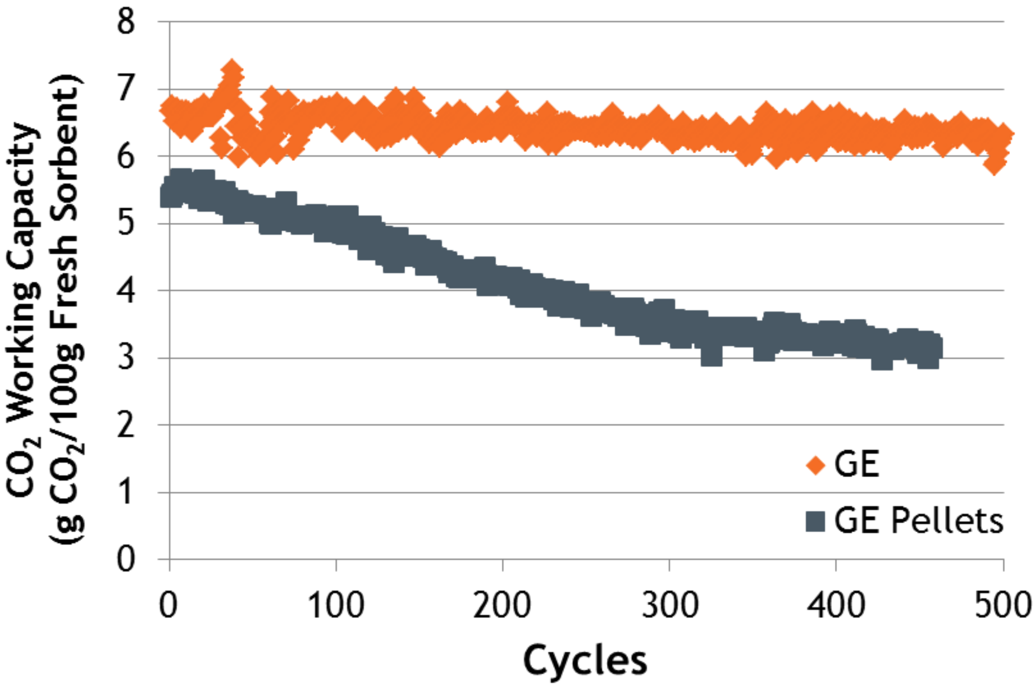
Attrition of GE-pellets (ASTM E728-91 R97)

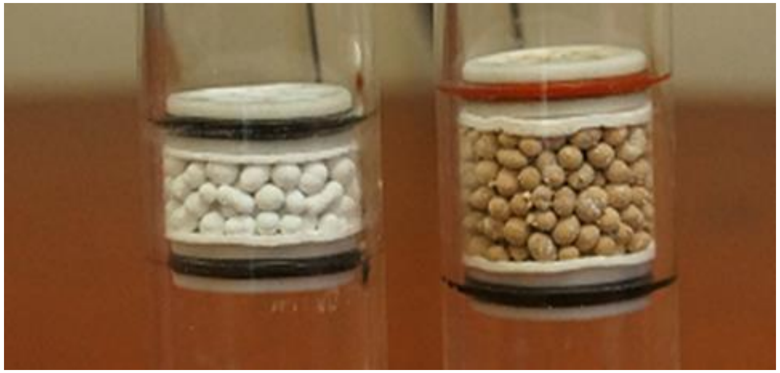


- Test conditions: 1 inch diameter and 2 ¼ inches long cylinder loaded with GE-pellets and 6 steel balls (¼ inch) was placed vertically in the rotary shaft of the ball mill to provide rotation along the length axis. The rotational speed of the ball mill was 60 rpm.
- The sample lost 2.5 % weight during the first 5 hours due to attrition and reached a plateau after 7 hours, for a total loss of **2.62 %**.

Phase II SBIR Results

Cycling stability of GE pellets





Left: GE Pellets, Right: GE pellets after 450 cycles of testing

- The cyclic stability of sorbent GE greatly decreased when the sorbent was formed into pellets
  - About 40% loss of initial delta CO_2 working capacity after 450 cycles

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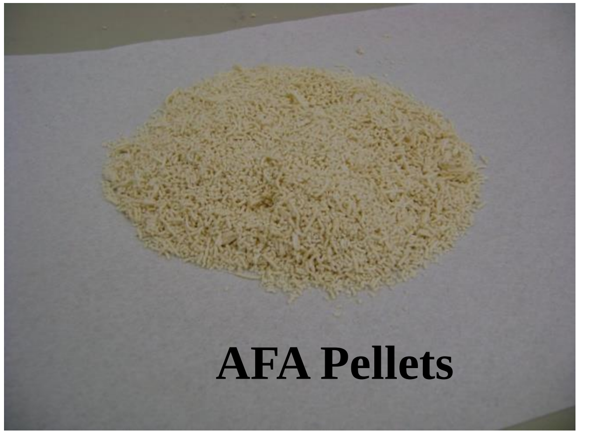
Phase II SBIR Results

AFA bench scale up production

- Fabricated > 90 lbs. of AFA (GE material) for cold fluidized bed testing.
- AFA sorbent (90 lbs.) was ground to < 106 um for pelletization (Pin-type impact mill equipment, Simpactor, was used at Sturtevant, Inc.).
- Sorbent was pelletized at University of Akron
  - Rod form, 3 – 5 mm long and 0.5 mm diameter, density ~ 1.3 g/cc
- Pellet grinding repeated to desired particle size (275 μm) for cold fluidized bed testing.



AFA Pellets

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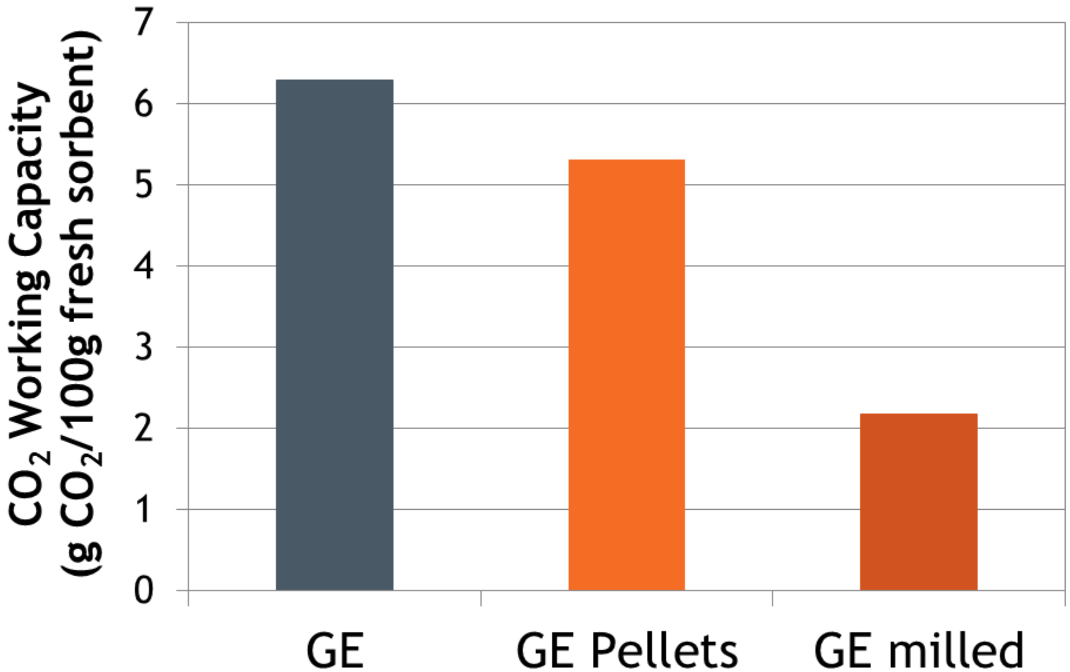
Phase II SBIR Results

Sorbent evaluation: GE –GE pellets – GE milled



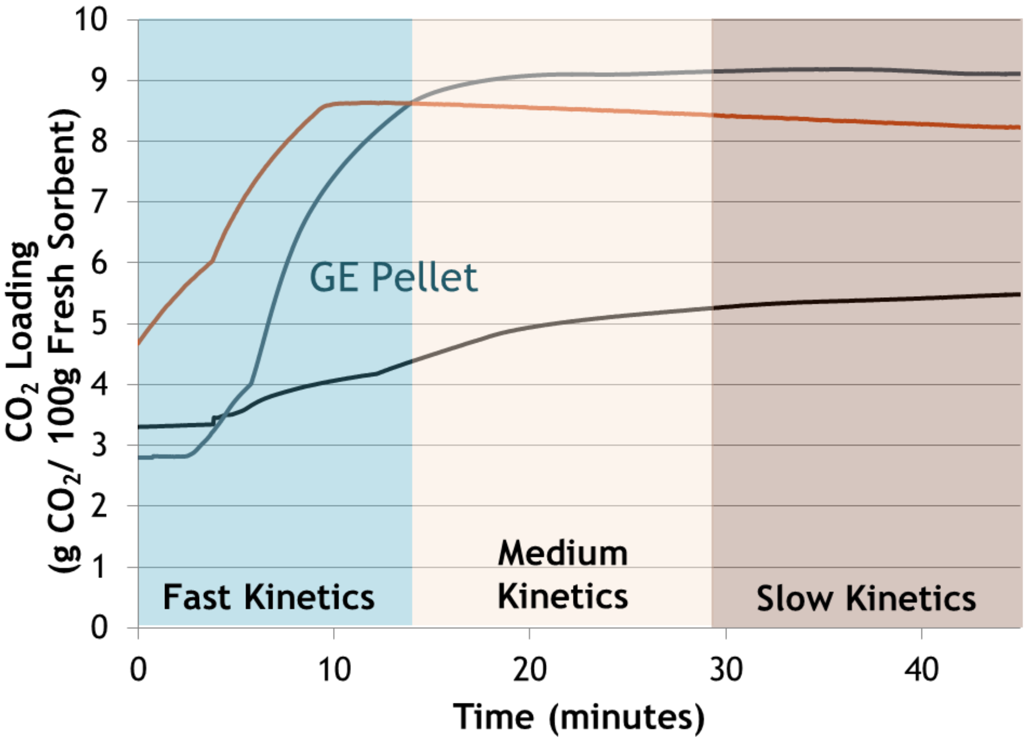
- CO_2 working capacity showed modest decrease when GE was formed into pellets

- CO_2 working capacity decreased when GE pellets were milled.

Phase II SBIR Results

Sorbent evaluation: GE –GE pellets – GE milled

Adsorption Conditions:
40°C, P_{CO2} = 0.15 bar



Time to 80% Capacity
GE – 4.6 min
GE Pellet – 9.7 min
GE Milled – 27.3 min

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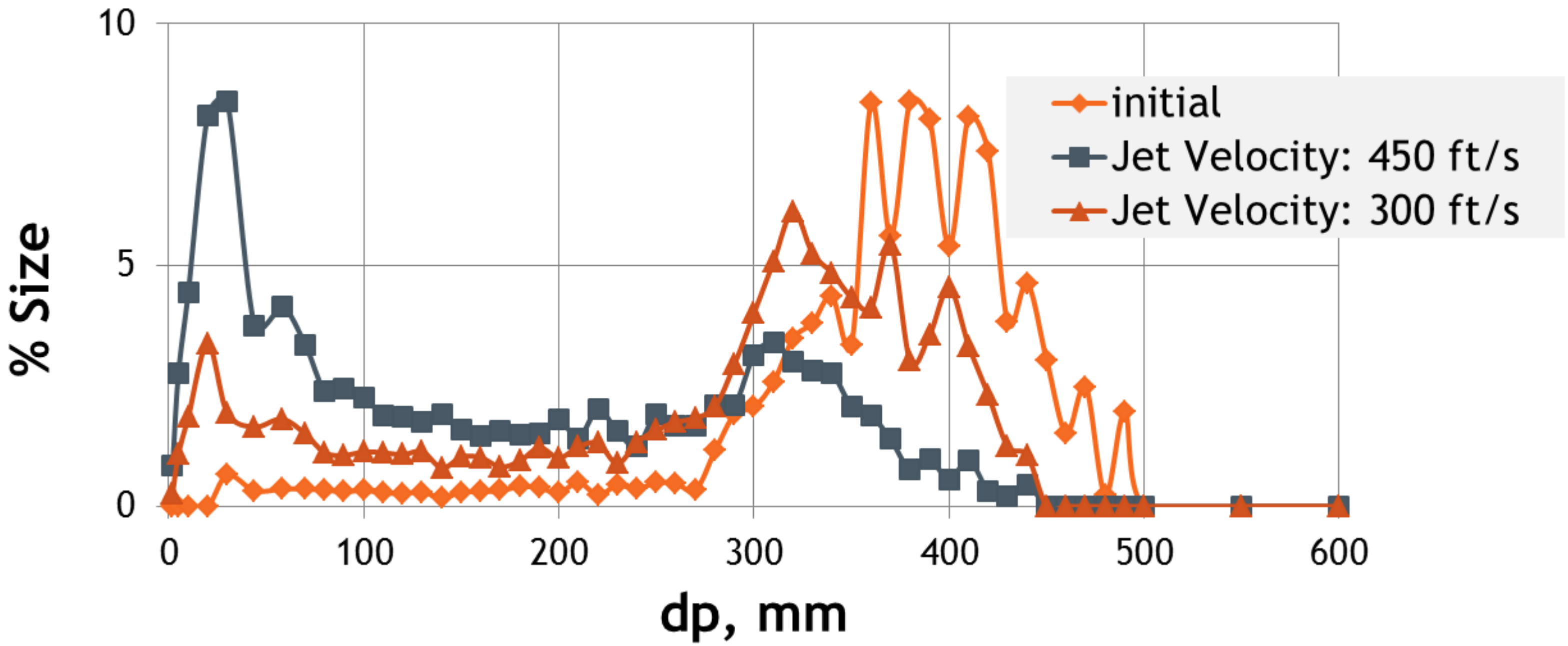
Phase II SBIR Results

Sorbent evaluation: GE milled (Attrition)

GE Milled showed higher attrition than the PSRI Standard FCC Eq.



Development required to reduce attrition

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Phase II SBIR Results

Physical Characterization of GE Milled

- Mean Particle Size (380µm)
- Particle Density (1.3109 g/ml)
- Explosion indices test (Kst value and Pmax)
  - explosive properties measured to quantify the severity of a dust explosion
  - Kst Value 29 bar*m/s
  - Max. Explosion Pressure 5.7 bar
- Crush Strength
  - Crush strength provides a quantitative measurement of particle hardness. However, it does not provide a means to directly predict attrition
  - On average 169g is needed to crush a ground GE pellet

Phase II SBIR Results

Key AFA Sorbent Characteristics

- **High CO_2 working Capacity**
  - Greater "working capacity" reduces required sorbent quantity, equipment size, and process costs
- **Kinetics**
  - Faster kinetics facilitates fast sorbent cycles, reducing sorbent quantity and potentially equipment size
  - Faster kinetics for CO_2 than H_2O can minimize impact of H_2O
- **Chemical Stability**
  - Minimal sorbent degradation for GE under process cycling conditions
- **Physical Stability**
  - Development required to reduce attrition

Phase II SBIR - Conclusions

- Over 200 Amine Functionalized Aerogel (AFA) sorbents screened and tested
- Three methods of amine incorporation were investigated.
- AFA sorbents have demonstrated:
  - ✓ *High CO_2 total capacity: ~ 22 wt% CO_2*
  - ✓ *High CO_2 working capacity: ~ 6.4 - 9 wt% CO_2*
  - ✓ *Good SO_2 resistance*
  - ✓ *Thermal stability across operational temperatures.*
  - ✓ *Superb stability : over 2,000 full sorption cycles*
- Produced large quantities of AFA sorbent
- Converted AFA powder into 4 cu. ft. of pellets for fluidized bed testing
- Performed fluidized bed testing

Amine Functionalized Aerogels are promising sorbents for CO_2 capture

Cooperative Agreement (CA) Program Overview

Award No. DE-FE0013127

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Program Objectives

Amine Functionalized Aerogel Sorbent

Form Pellets with Binder

<u>Develop Aerogel Sorbent at Bench Scale for CO_2 Capture</u>

- Improve Amine Functionalized Aerogels
- Develop Pellet Binder Formulations
- Develop Pellet forming process
- Develop SOx diffusion barrier
- Test & Evaluate Sorbent Technology at Bench Scale

Proprietary information has been deleted.

Program Team

Sorbent optimization – bench scale production

Sorbent testing – bench scale evaluation

Sorbent pelletizing – Sorbent flue gas poisoning optimization

Program Tasks

BP#	Task#	Description
	Task 1	Project Management
BP1	Task 2	AFA Sorbent Development
	Task 3	Sorbent Coating Development
	Task 4	Sorbent Evaluation
BP2	Task 5	Aerogel Bead Fabrication
	Task 6	Pellet Development
	Task 7	Pellet Evaluation
BP3	Task 8	Pellet Production
	Task 9	Fluidized Bed & Pellet Evaluation
	Task 10	Techno-Economic Evaluation
	Task 11	Environmental Health and Safety Evaluation

Task 1. Project Management

1. Refine Program Management Plan with Federal Project Officer - Complete
2. Program Management Plan Maintenance & Revision
3. Monthly Teleconferences
4. Budget Period Reviews

Task 2. Amine Functionalized Aerogels (AFA) Sorbent Development

- Optimize the 3 most promising AFA formulations:

Sorbent	Total CO_2 loading (wt.%)	CO_2 delta loading (wt.%)	Kinetics	Stability
GE	8	6.4	**Fast**	**High**
HQ	~ 15	11	**Medium**	**Low**
IJ	~ 22	12	**Slow**	**Low**

GE sorbent: mono-amine alkoxysilane functionalized aerogel

HQ sorbent: polyimine loaded hydrophobic aerogel

IJ sorbent: polyamine alkoxysilane terminated functionalized aerogel.

Task 2. AFA Sorbent Development

- Amine concentration will be maximized by varying components of the aerogel formulation.
- Qualitative optimization of these materials will be dedicated to:

  1) Maximizing the CO_2 capacity.

  2) Improving resistance to flue gas contaminants.

  3) Maintain kinetics for realistic fluidized bed operation.

  4) Maintain high cyclic adsorption stability.

Task 3. Sorbent Coating Development

- Perform CO_2 capture performance test screening on AFA sorbents fabricated during Task 2.
- Develop an efficient low-cost coating technology (compatible with AFA sorbent)
- Evaluate AFA resistance to performance degradation in the presence of NO_x and SO_x.
  - Using simulated flue gas
  - Determine CO_2, NO, NO_2 and SO_2 breakthrough curves during adsorption
  - Calculate adsorption kinetics, and adsorption equilibrium loading
- Sorbent structural properties and elemental composition will be determined.

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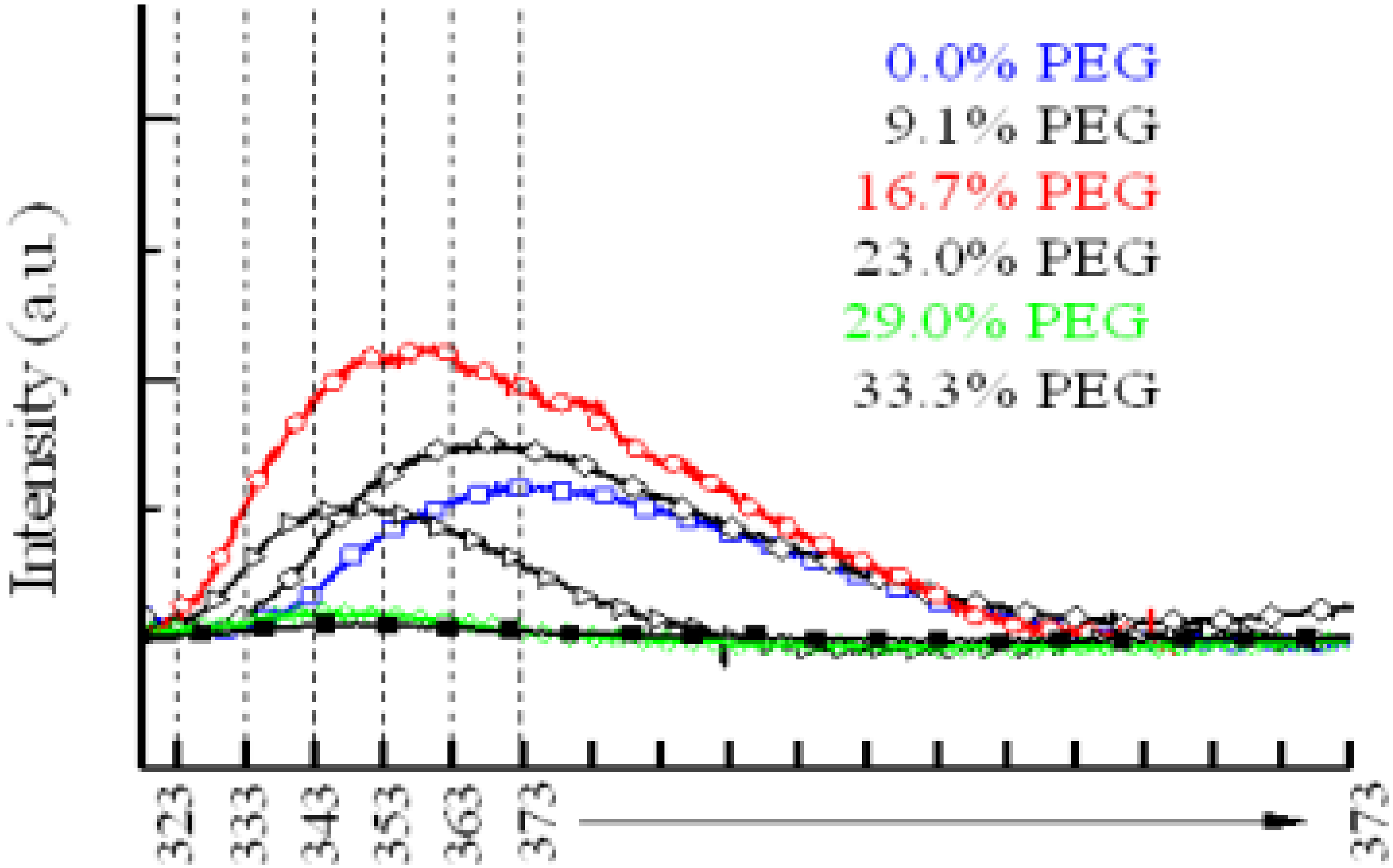
Task 3. Sorbent Coating Development

- Temperature-programmed desorption of CO_2 from mixtures of TEPA and PEG



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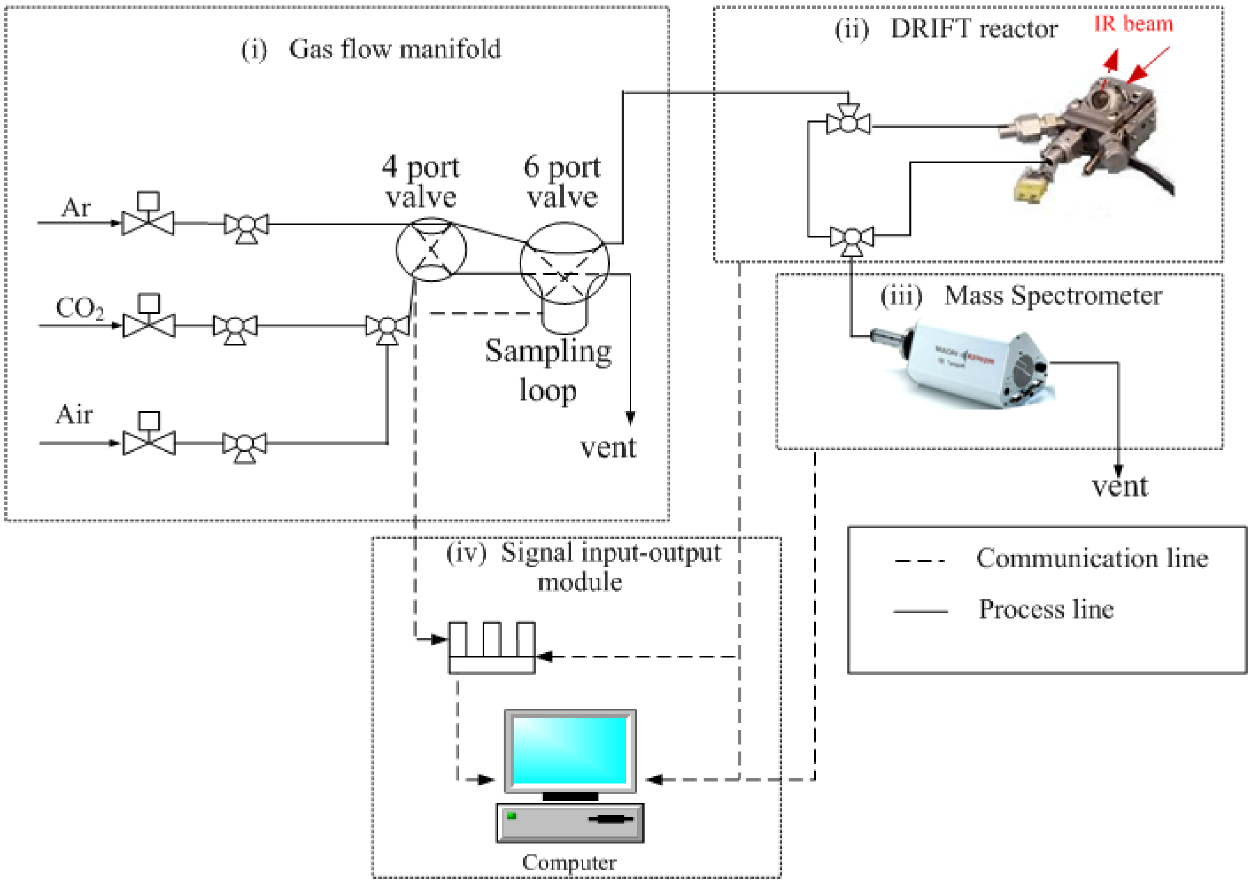
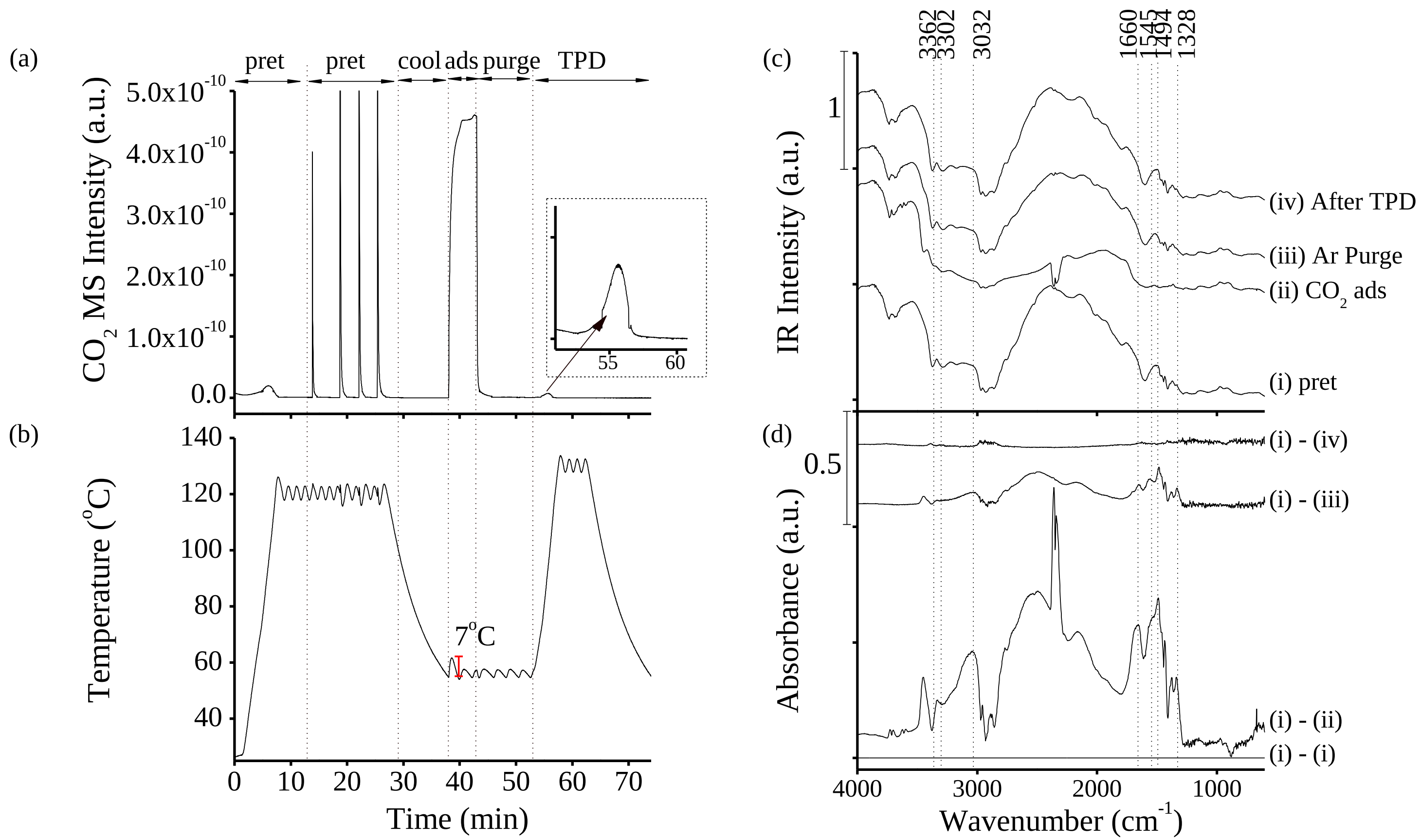
In-Situ Infrared Studies

- Experimental apparatus for in-situ DRIFTS used in CO_2 capture study.



Mass Spectrometry

- CO_2 MS Intensity (m/e=44), temperature, single beam and absorbance spectra during a typical CO_2 capture cycle.



Task 4. Sorbent Evaluation

- Initial evaluation of CO_2 loading on a sorbent via thermogravimetric analyzer (TGA)
- Assess the H_2O loading of the sorbent at select temperatures and H_2O partial pressures using a TGA
- Develop CO_2 loading isotherms over a range of temperatures and pressures
- Assess selectivity of sorbent for CO_2 and any negative impacts from common flue gas constituents using fixed-bed coupled with mass spectrometer
- Assess the longer-term stability of the sorbent when exposed to typical flue gas constituents using an automated fixed bed

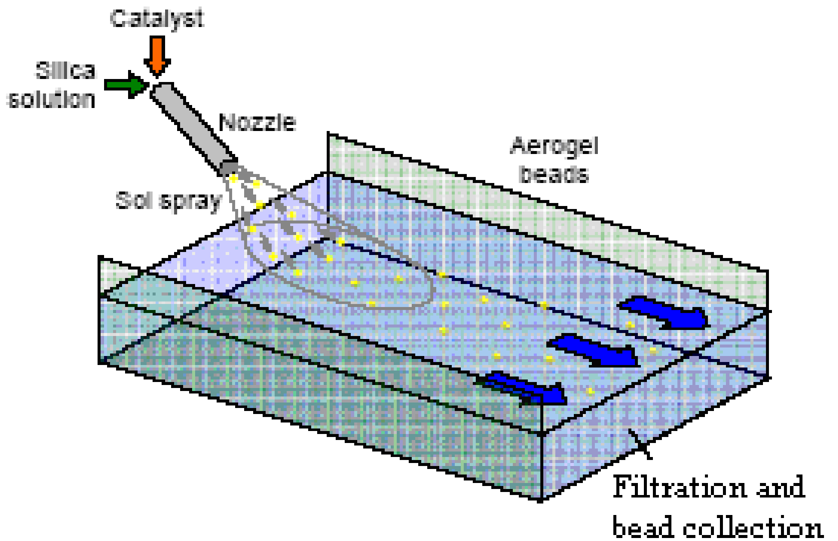
Task 5. Aerogel Bead Fabrication

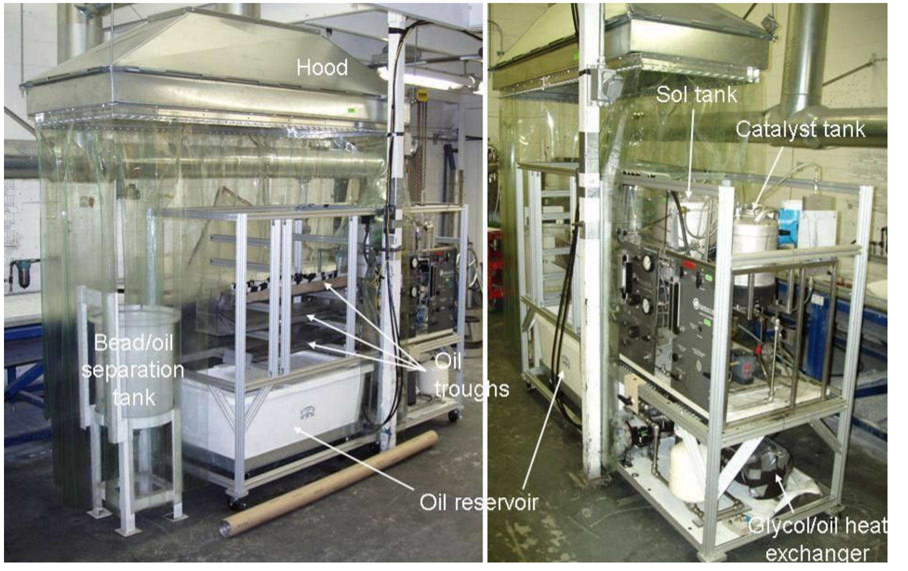
Objective: *Produce AFA sorbent in bead form and determine if the pelletization step can be eliminated while maintaining adequate sorbent performance.*

- AFA bead performance will be compared directly to those of AFA pellets formed using the method from UA.
- The CO_2 capacity, cycle life, density, attrition index and crush strength will be compared.

Task 5. Aerogel Bead Fabrication

An aerogel bead-fabrication process has been developed previously by Aspen.





- Aspen will produce the sorbent beads in a range of particle sizes to be assessed as a CO_2 sorbent for operation in a fluidized bed.
- Aerogel beads will be sent to ADA and UA for CO_2 performance evaluation and for addition of SO_2 resistant coatings.

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Task 6. AFA Sorbent Pellet Development

- UA pellet forming process will be used to fabricate AFA pellets.
- Parameters to optimize:
  - Binder – Aerogel compatibility
  - Binder/aerogel ratio
  - Pellet size
  - Binder concentration, impregnation time, curing.
- Aspen, UA will tailor sorbent to have optimal diameter and density for ADA fluidized bed.

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Task 7. Pellet Evaluation

- Follow procedures for powders.
- Crush strength and attrition resistance
- Assess the H_2O loading of the sorbent at select temperatures and H_2O partial pressures using a TGA
- Develop the CO_2 loading isotherms over a range of temperatures and pressures *(2 samples)*
- Assess the selectivity of the sorbent for CO_2 and any negative impacts from common flue gas constituents using a fixed-bed coupled with a mass spectrometer
- Assess the longer-term stability of the sorbent when exposed to typical flue gas constituents using an automated fixed bed

Task 8. Bench Scale Pellet Production

- Aspen will produce larger batches (2 pounds each) of the optimized AFA.
- Large capacity extractors will be used for the production.
- Aspen will demonstrate the consistency of the quality of AFA batches.
- Different AFA batches will be subjected to analytical testing
  - Surface area, XPS, particle size measurement
  - CO_2 capture evaluation
  - Stability over 250 cycle.
- AFA batches will be converted to pellets using optimum pelletization conditions (binder mixing, pellet forming, treating with SO_2 resistant coatings).
- The pellets grinding conditions will be optimized to desired particle size for fluidized bed tests. Sieving may be required to reduce fines.

Task 8. Bench Scale Pellet Production

Amine-grafted sorbent (powder)

Add ← Binder solution

Mix at 25 °C

Wet paste

Wet extrusion (1 or 2 mm die)

Wet extrudate → Coating with powder sorbent

Drying at 130 °C

Cutting

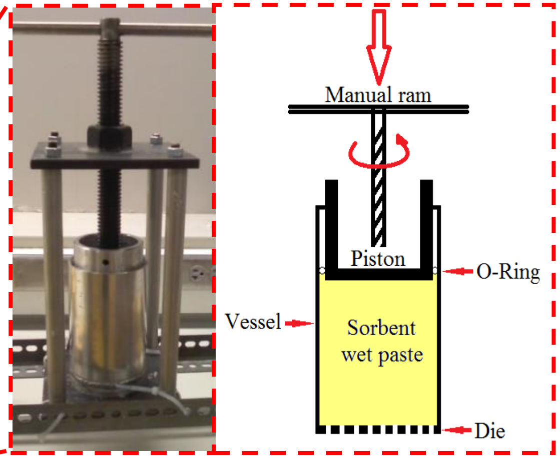
Rod pellets

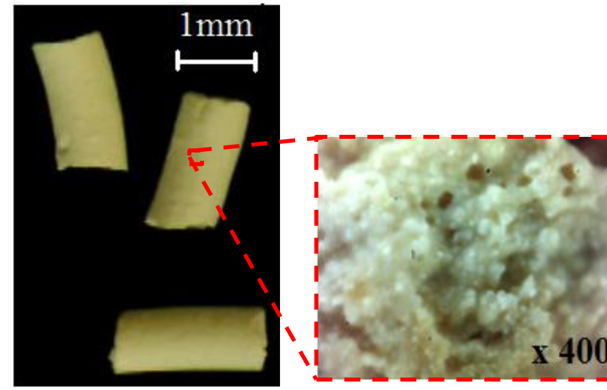
Coating with powder sorbent

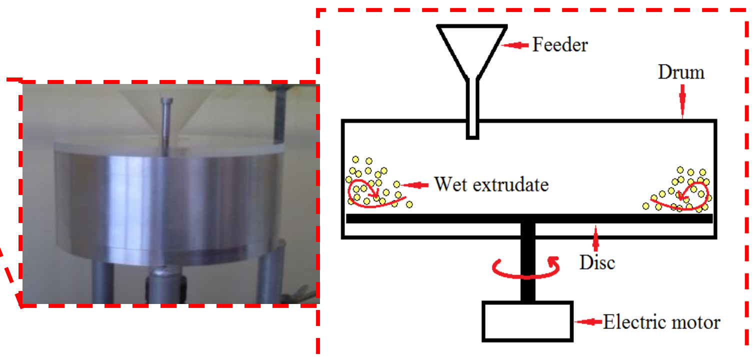
Spheronization

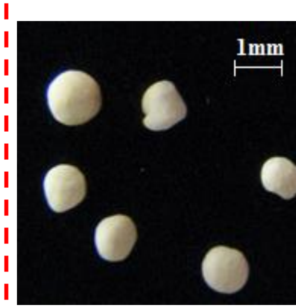
Drying at 130 °C

Spherical pellets







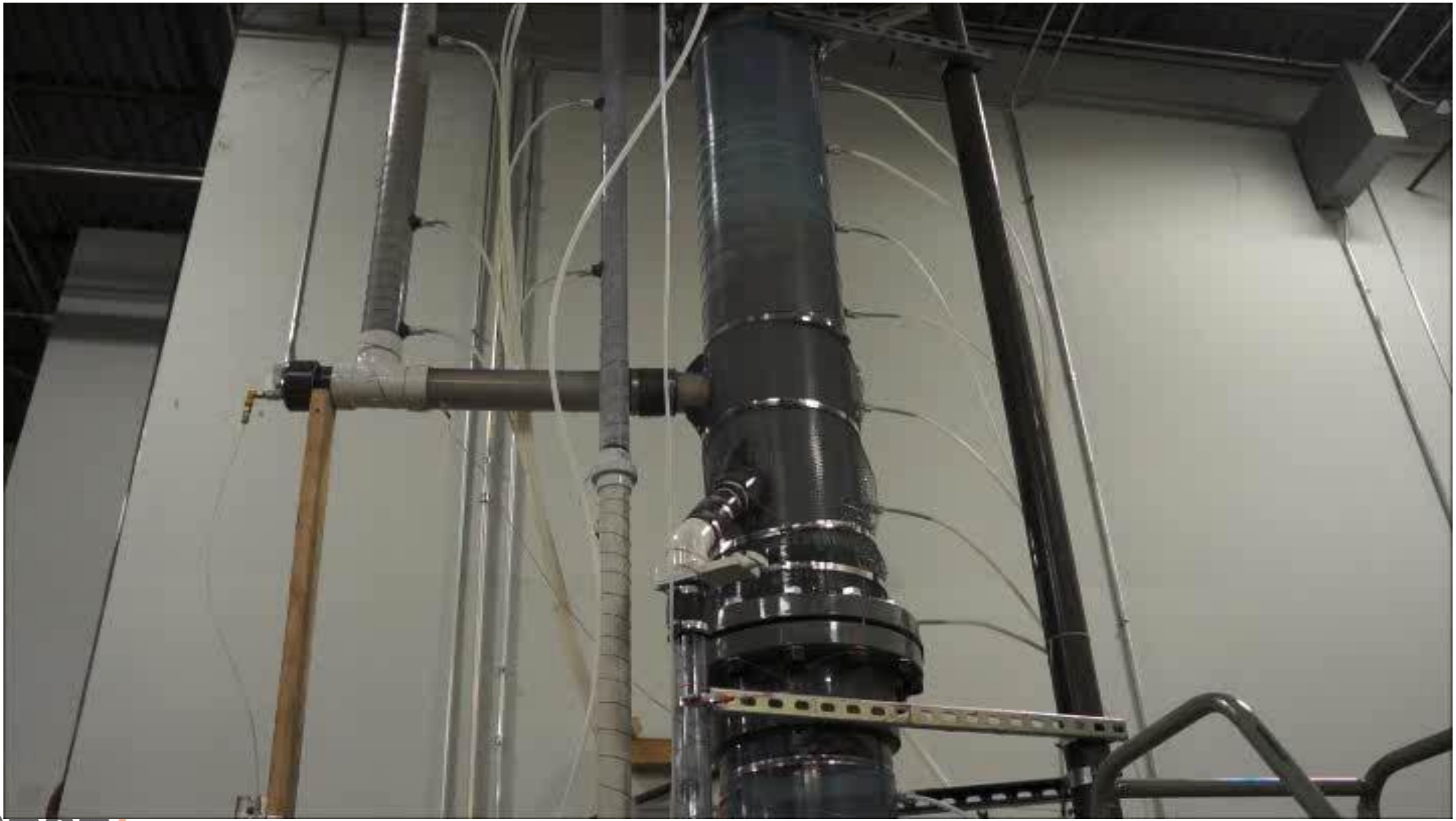


Task 9. Cold Fluidized Bed & Pellet Evaluation

- Testing with Cold Flow Models
  - Optimal particle size distribution
  - Fluid bed density
  - Fluidization regime (e.g., bubbling, slugging, fast fluidization, etc.) at different gas velocities
  - Gas velocity required to achieve the desired fluidization regime
  - Quality of fluidization determined both visually, and by means of high frequency ΔP bed fluctuation measurements
  - Bubble volume fraction
  - Heat Transfer Coefficient

Bubble Flow Regime

Slug Flow Regime



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Turbulent Flow Regime

Task 9: Pellet Evaluation

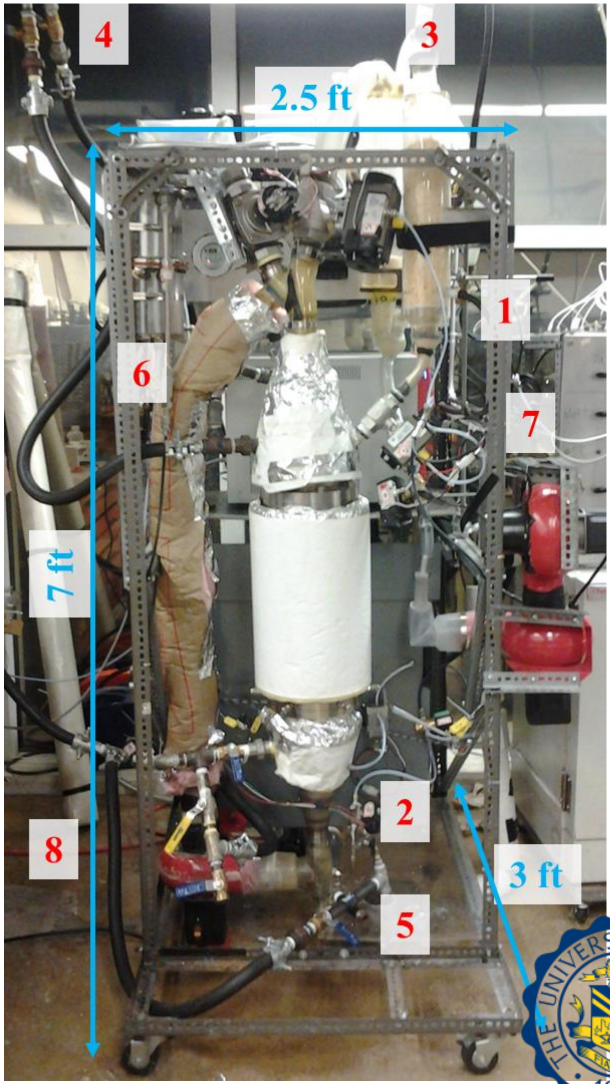
- Physical Characterization of Pellets
  - Specific Heat Capacity
  - Heat of Reaction
  - Particle Density
  - Particle Size
  - Jet-cup attrition Testing

Task 9. Fluidized Bed & Pellet Evaluation

Akron system evaluates CO2 adsorption/desorption at bench scale

10 kg Circulated Bed CO_2 Capture Unit

- Height: 7 ft
- Width: 2.5 ft
- Depth: 3 ft
- Weight: 300 lb (with frame)



Gas connections

1.Flue gas inlet (20 lpm): 1 x ¼”

2. CO_2 purge inlet (6 lpm): 1 x ¼”

3. Outlets (vents): 3 x ¾”

Utilities connections

4.Saturated steam (60 psi): 2 x ¾”

5.Injection steam* (130 ^oC): 1 x ¼”

6.Cooling water (15 lpm): 1 x ¼”

7.Control valves air (70 psi): 6 x ¼”

Electrical requirements

8. AC electrical connections: 110/120V

*Injection steam <u>must be</u> superheated and Cu-free.

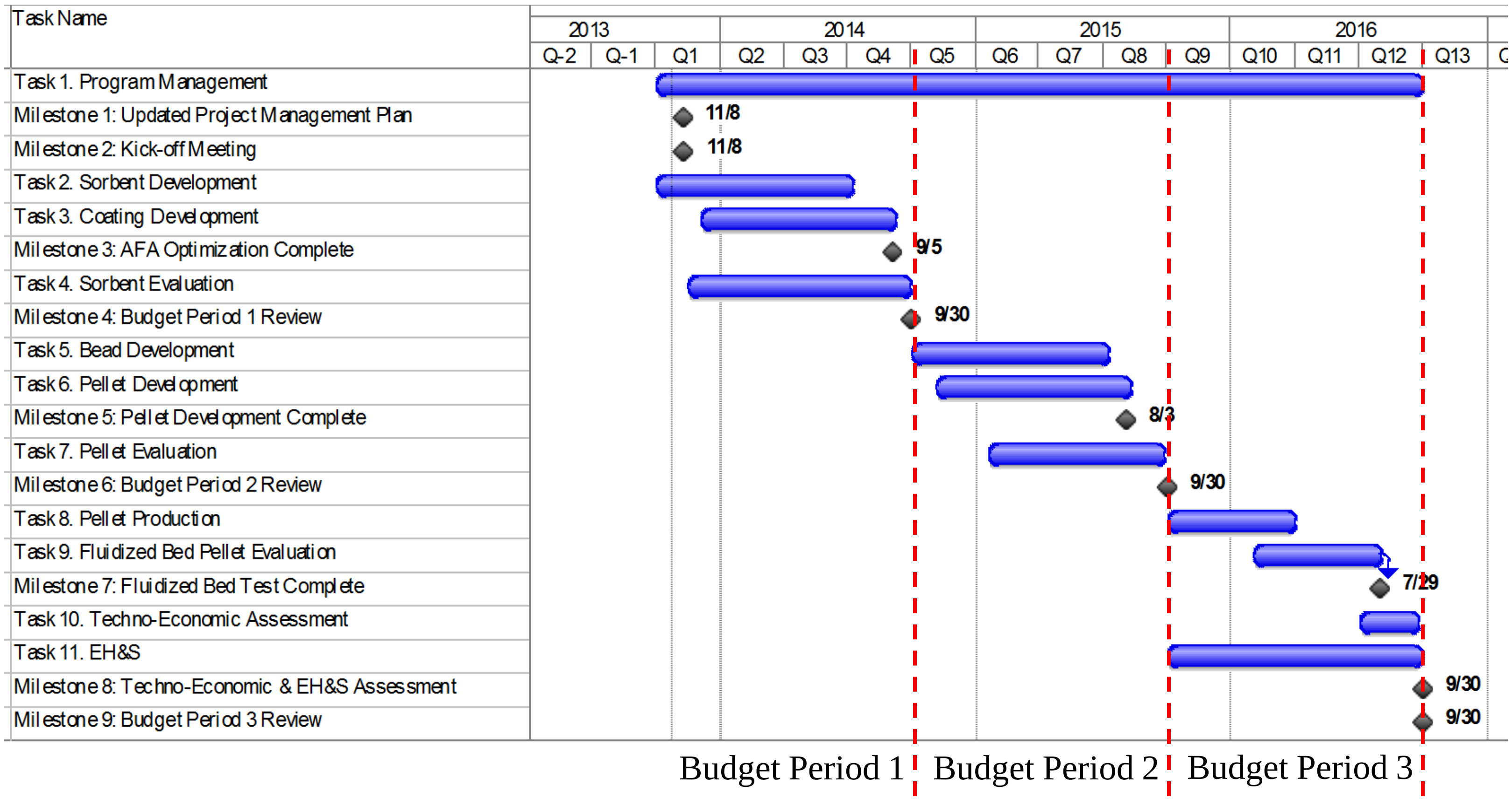
Task 10. Techno-Economic Evaluation

- ADA, through a separate DOE contract, has developed and will be refining a techno-economic assessment of their fluidized bed capture process using a single sorbent
- Techno-economic assessment includes factors such as optimal operating conditions for adsorption and regeneration, sorbent CO_2 working capacity, sorbent stability, resistance to acid gases, sorbent cost, sorbent attrition rate, and several other key parameters
- Techno – Economic Assessment will be reported once the Cold-Flow Model data is finalized

Task 11. Environmental Health and Safety Evaluation

- Potential risks related to the process of AFA manufacturing.
- Potential toxological risks related to manufacture of AFA sorbent.
- Assessment of physical and equipment risks by handling the AFA sorbent.
- The compliance and regulatory implications of the technology.
- Safe handling and safe storage of AFA sorbent.
- Process optimization study to minimize use of toxic/hazardous substances.

Program Schedule/Milestones



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Program Deliverables

1.	Kickoff Meeting	Presentation
2.	Topical Report BP1*	Document
3.	Quarterly Reports	Document
4.	Budget Period 1 Review	Presentation
5.	Sorbent Production	E-mail confirmation
6.	Topical Report BP2**	Document
7.	Budget Period 2 Review	Presentation
8.	Technical & Economic Analysis Complete	T&E Document
9.	EH&S Assessment Complete	T&E Document
10.	Final Scientific Report***	Document
11.	Budget Period 3 Review	Presentation

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Current status

Task 2: AFA Sorbent Development

- Initiated optimization of the three best formulation developed during the Phase II program
  - Increase the amine loading (primary amine-alkylsilane precursor) in AFA sol-gel formulation
  - Improve the thermal stability of Polyimine (or Polyamine) loaded AFA sorbent
  - Investigate two different routes of amine impregnation (and grafting) onto hydrophobic silica aerogel structure

- A few samples have been fabricated and they will be sent to ADA for CO_2 capture performance.

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Thank You

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